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# ABSTRACT

Among the problems associated with long term high temperature service of hybrid material systems is interdiffusional compatibility of the system's component materials. Efforts in this program have focussed on several areas of this problem. One of the areas being examined is the use of alkali and alkali earth metals for diffusion barriers. These barriers are being applied by ion implantation. The model system being used is the W/Ni system. Preliminary results of barrier effectiveness are given. Also being studied are the interdiffusional kinetics of metal/intermetallic and intermetallic/intermetallic ternary systems. Hybrids being examined include W/Ni<sub>3</sub>Al and Ni<sub>3</sub>Al/TiAl.

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## I. INTRODUCTION AND BACKGROUND

In many structural applications, conventional materials can no longer fulfill the increasing drastic service demands and/or long-term reliability requirements of future high temperature components. High temperature material systems for Air Force jet engine applications are especially prone to increasingly rigorous high temperature and life-cycle demands. In some respects the demands are a result of new designs that will improve product performance. For example, higher operating temperatures of jet and rocket engines increases the thermodynamic efficiency and power output, while lowering both fuel consumption and exhaust emissions.

At present gamma/gamma prime strengthened superalloy single crystals place a ceiling on the operating turbine blade skin temperature of jet engines at approximately 1050°C [1,2,3]. The need to push this operating temperature limit higher, and also increase the material reliability at current operating temperatures, has resulted in research attention redirected from conventional alloys to more novel materials concepts like structural ceramics, carbon-carbon systems, and such hybrid materials systems as oxide dispersion strengthened alloys and fiber reinforced alloys, intermetallics, and ceramics. This program addresses hybrid materials systems for prolonged high temperature applications.

Hybrid materials systems intended for ambient temperature or mid-range temperature service require a certain degree of mechanical property compatibility between the system components. For example, metal/metal or graphite reinforced epoxy composites

require ductile matrix behavior and a strong fiber/matrix interface in order to ensure adequate load transfer from the matrix to the fiber components. For hybrid material systems intended for repeated thermal excursions to high temperatures, thermal-mechanical compatibility is also required. The situation becomes even more complex for hybrid systems that are subjected to prolonged high temperature excursions. In addition to mechanical and thermal-mechanical compatibility, time-reliable hybrid materials system components must also exhibit chemical compatibility between the composite components at the intended service temperatures.

Thermal-mechanical compatibility is influenced by the thermal expansion among the composite components, the thermal conductivity of the hybrid components, and the ability of the components to accommodate strains associated with thermal cycling. The high temperature chemical compatibility is related to such thermally activated processes as interdiffusion and intermediate phase reaction kinetics.

In general, the thermal-mechanical compatibility of hybrid and composite systems is well understood. For example, many researchers have used elasto-plastic analysis to describe and/or measure the effect of thermal mismatch in hybrid and composite systems [4-8].

A difference in the chemical potential between hybrid components can result in interdiffusion among the components provided sufficient thermal activation exists. High temperature chemical compatibility becomes increasingly important for higher operating temperatures and prolonged exposures. Interdiffusion

related phenomena that can affect composite performance include the growth of intermediate and potentially brittle phases at the component interfaces, dissolution of the reinforcing fibers, and matrix poisoning of the fibers. Such interactions can result in time limited service constraints.

Current theoretical or analytical models are in the rudimentary stages with respect to predicting phase stability in complex systems and in forecasting the degree of interdiffusion and its effects. As a result, predictive chemical compatibility between hybrid components can only be made on a restrictive basis. Actual confirmation of the compatibility and the amount of change has to be made experimentally.

Interdiffusion controlled reactions in high temperature hybrid materials systems have been reported in the literature with respect to such constitutionally simpler (metal/metal) hybrid systems as the tungsten fiber reinforced superalloys (TFRS) [1,9-30]. Much of this literature has Russian origins. The current state of understanding can be compacted as follows: (1) W-fiber and matrix recrystallization due to the interdiffusion (i.e. poisoning), (2) W-fiber dissolution, and (3) intermetallic phase growth at the fiber/matrix interface. In addition, it should be noted that even in systems involving alloys strengthened by the so-called inert oxides, the temperature ceiling of use is controlled by oxide coarsening [31] and by the formation of voids resulting from unbalanced interdiffusion [32].

## II. CURRENT STATUS

Our research efforts during the first renewal period of this grant have been focussed on two separate areas of composite interface technology. One phase of the current research is devoted to the investigation of diffusion barriers which will inhibit interdiffusion between the reinforcing and matrix materials across fiber/matrix interface. The second, parallel, effort is evaluating the interdiffusional stability and chemical compatibility of selected metal/intermetallic and intermetallic/intermetallic composite systems.

In response to the problem on interfacial compatibility of composite constituents we are investigating the effectiveness of an ion implanted diffusion barrier which shows promise. In order to test the feasibility of this barrier layer we have chosen the simple W / Ni system as a model. This system was chosen due to the well understood interdiffusional phase stability and kinetics of interdiffusion. The barrier which we are primarily interested in is one of the alkali metal ions. Alkali ions that were originally considered were Na, Ca, Ba, and K. These alkali and alkali earth metals possess very large molar volumes. See Table I. It might be expected that by incorporating these atom in a thin layer at or near the interface that diffusional behavior may be modified due to the internal strain caused by these atoms in the lattice. However, if excessive strain is caused the lattice may recover through thermally activated processes and the additions may be ineffectual or even intensify interdiffusion. Diffusion couples incorporating these layers to evaluate their feasibility have been fabricated.

TABLE I.

Elemental Molar Volumes (cm<sup>3</sup>)

Ni	6.6
W	9.5
Na	29.9
Ca	32.7
Ba	39.0
K	45.0



Diffusion couples have been made by ion implanting alkali ions into a tungsten substrate and subsequently ion beam sputtering a film of nickel over the implanted surface. Conditions which were utilized for this process are shown in Table II. The nickel films were found to exhibit the typical sputtered microstructure of a thin epitaxial layer, followed by a region of fine equiaxed grains, with the remainder being long columnar grains oriented in the growth direction. These diffusion couples were annealed at 1500K in vacuum for 50 hours.

Composition--position plots were obtained for the couples using a calibrated EDS microprobe. Concentrations were determined at 1 micron intervals across the interdiffusion zone. An example of such a plot is shown in Figure 1. Due to the rather long time required to deposit the nickel film on the tungsten substrate, combined with the nickel's microstructure, a large degree of grain boundary diffusion is evident.

This consequence of the relatively simple fabrication technique employed limits the analysis of these couples to a comparative analysis of the extent of interdiffusion between the different barriers employed as well as the control specimen without any barrier. Figures 2 and 3 illustrate the effect of the individual barrier atoms relative to the couple without any barrier and the couple initial condition. As can be seen, calcium atoms were found to have a seemingly pronounced effect on reducing interdiffusion in the W/Ni system while sodium was found to have a negligible effect the interdiffusional penetration. In fact, preliminary data suggests that potassium may even promote accelerated diffusion for the limited conditions tested to date.

TABLE II.

Ion Implantation Conditions

Ion Density	$6.4\text{E}-11 \text{ cm}^{-2}$
Accelerating Potential	190 kV
Median Penetration	250-350 nm

Ion Beam Sputtering Conditions

Substrate Temperature	400°C
Deposition Time	70 Hours

FIGURE 1

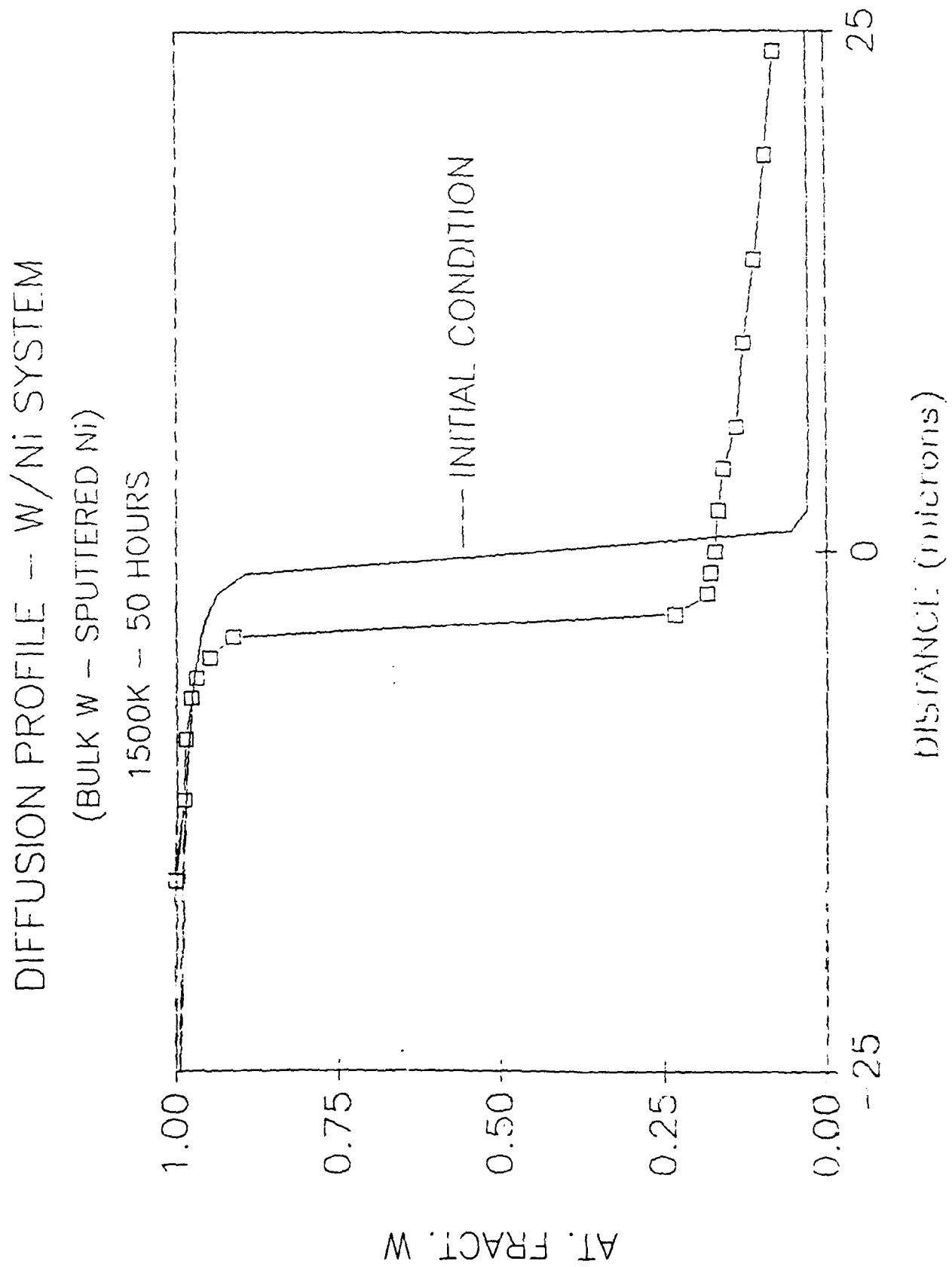
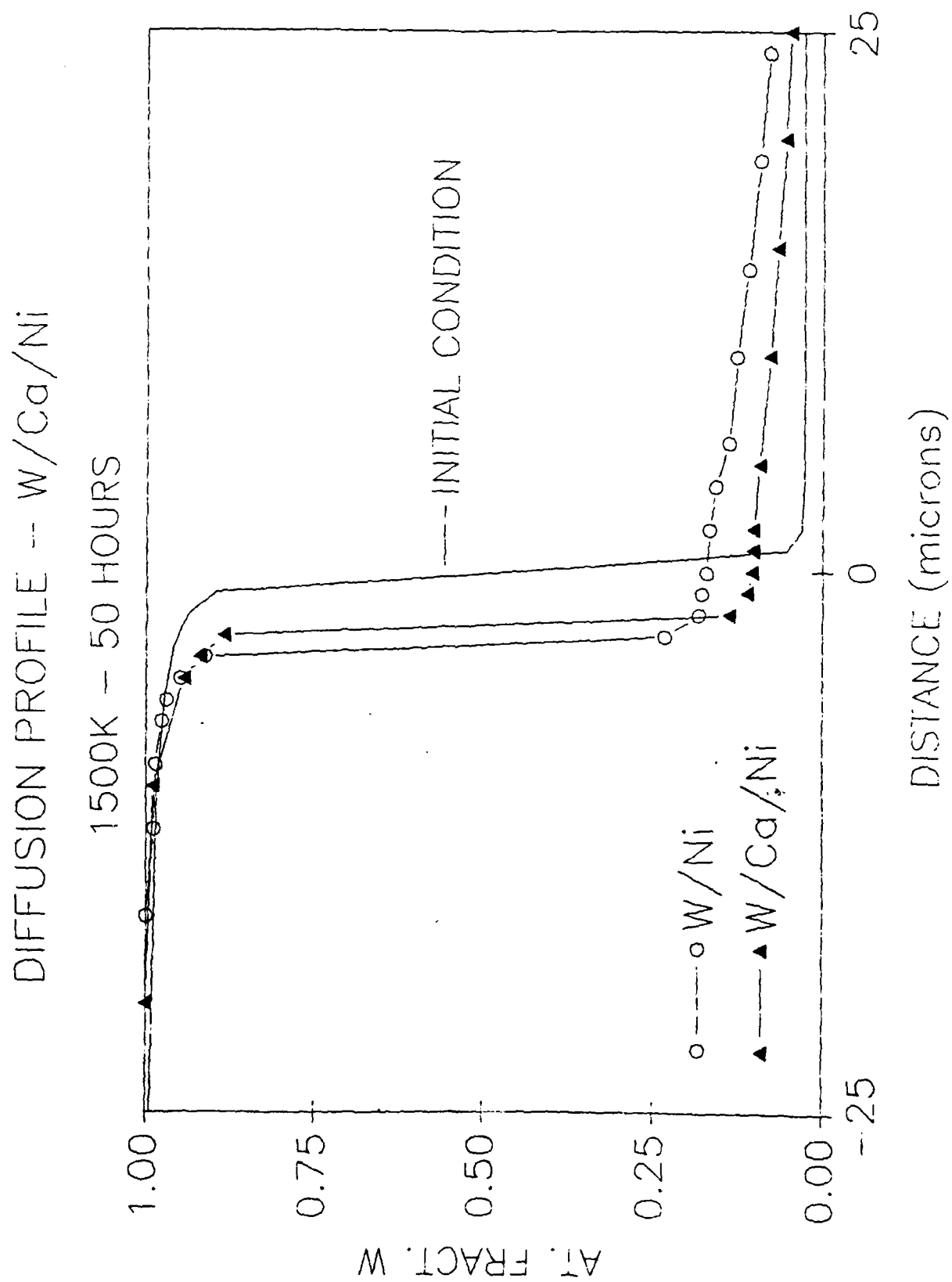


FIGURE 2



# DIFFUSION PROFILE -- W/Na/Ni

1500K - 50 HOURS

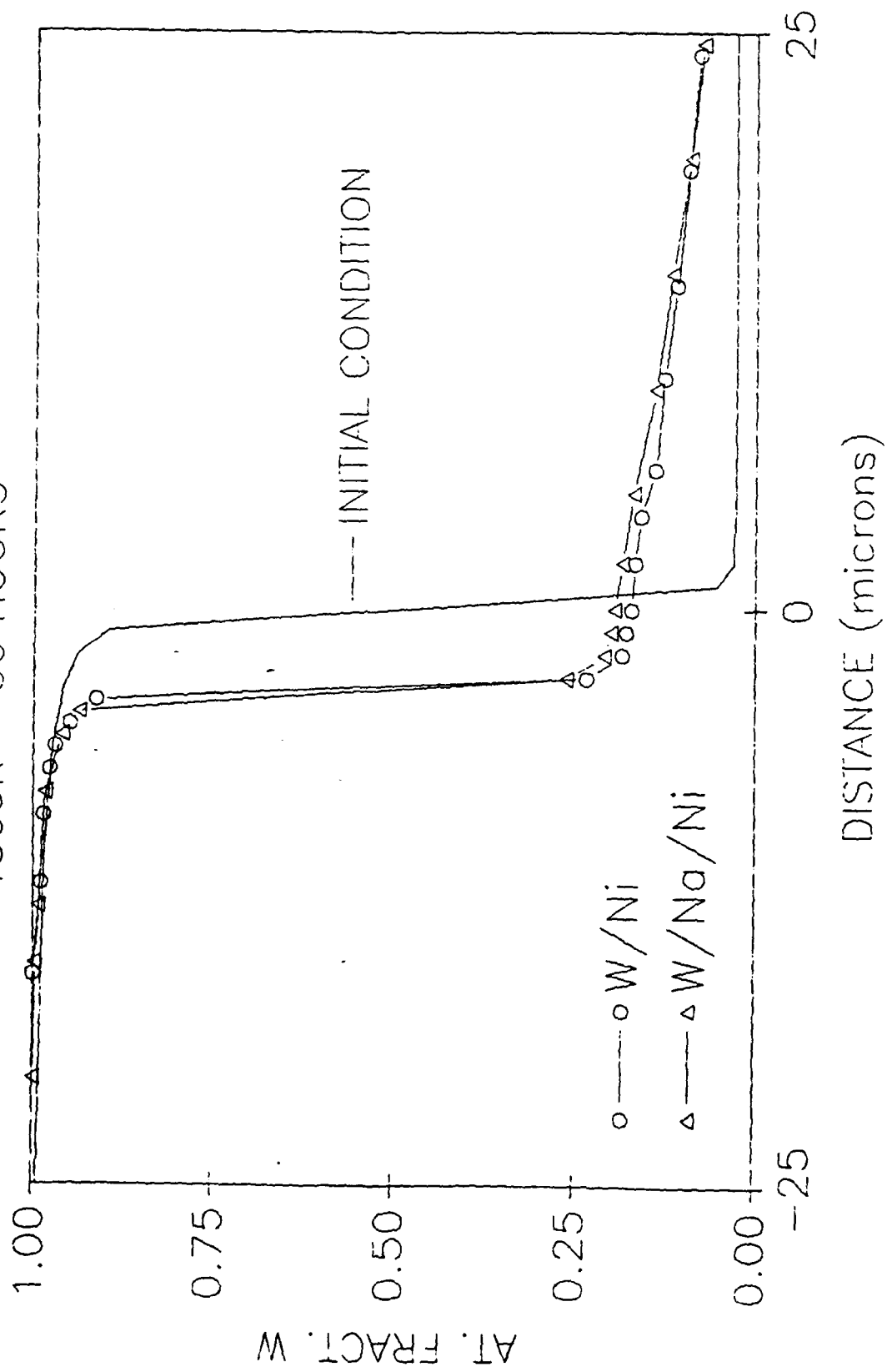


FIGURE 3

This may be due to excessive local damage caused by the implantation of too many of these large atoms in too thin a surface region.

In order to confirm the effect of the implanted calcium barrier in the W / Ni system, four new sets of diffusion couples are currently being fabricated. Since quantitative analysis of the original couples was impossible due to the microstructure of the sputtered Ni film, these new diffusion couples are being made with bulk W and Ni sheet. We have also limited ourselves to the single barrier that showed promise based on the original data, namely the Ca ions. The surface of the W substrates have been implanted with three different doses of the Ca ion:  $5 \times 10^{12}$  ions/cm<sup>2</sup>,  $5 \times 10^{13}$  ions/cm<sup>2</sup>, and  $5 \times 10^{14}$  ions/cm<sup>2</sup>. These implanted surfaces are currently being diffusion bonded to the Ni sheet by a hot-pressing technique. This hot pressing is being done free of charge by the Rockwell International Science Center. Additional couples without a barrier are also being fabricated to serve as control samples. Once these couples are fabricated, they will be annealed in vacuum at three temperatures for three different times. This test matrix is shown in Table III. After annealing, composition-position profiles will be obtained by EDS, and diffusion coefficients will be extracted via Boltzmann-Matano analysis.

This quantitative analysis will allow us to ascertain several important features of the calcium barrier. First, within each annealing temperature, the effect of barrier dose may be evaluated in order to better optimize the barrier dose needed. Second, the effect of temperature on the barriers will be

TABLE III.

Diffusion Barrier Test Matrix

Temperature	Time
1300K	25 Hrs.
	100 Hrs.
	225 Hrs.
1400K	25 Hrs.
	100 Hrs.
	225 Hrs.
1500K	25 Hrs.
	100 Hrs.
	225 Hrs.

ascertained. Third, information on the effect of time at temperature on the effectiveness of the barriers will be extracted. Thus, information on the overall barrier effectiveness, as well as keys for barrier optimization, will be determined.

As previously mentioned, these diffusion couples are currently being hot-pressed and we expect to be able to begin the specimen annealing in January.

Our efforts which have addressed the compatibility of metal/intermetallic and intermetallic/ intermetallic composite systems have focussed on the W/Ni<sub>3</sub>Al and Ni<sub>3</sub>Al/TiAl systems. These systems have been chosen for several reasons. Although these systems show promise from an engineering viewpoint due to the availability and properties of commercial tungsten fibers and the expected oxidation resistance of aluminide matrix materials, perhaps more importantly, they enable a fundamental scientific analysis. By limiting the analysis to ternary systems (for both metal/intermetallic and intermetallic/intermetallic couples) the analysis of the interdiffusional kinetics and intermediate phase formation is greatly simplified. As a consequence of this simplification, results for metal/intermetallic and intermetallic/intermetallic diffusion may be compared and incorporated into a model to describe universal behavior in ternary systems. Several avenues of analysis are promising. For the simpler W/Ni<sub>3</sub>Al system, a first effort of Boltzmann-Matano analysis may be easily employed by considering the system a pseudobinary and considering the diffusion path constant with temperature. Thus, the



interdiffusional kinetics may be expressed in terms of W concentration and temperature in a simple fashion. The correlation of the resultant composition dependent interdiffusion coefficients with ordering parameters, as well as kinetics of formation of any intermediate phases ( $\text{Ni}_3\text{W}$ ,  $\text{Ni}_4\text{W}$ ), should any form, should indicate what, if any, more complicated analyses might be called for. Possibilities include the cluster variation method, the path probability method, and Monte Carlo techniques.

We encountered some problems with startup in this area of the program. Although  $\text{Ni}_3\text{Al}$  was rather easily acquired,  $\text{TiAl}$  was difficult to obtain. An industrial source was finally found to EB melt a button for our use but this has delayed progress in our intermetallic/intermetallic efforts. There has also been some delay in fabricating the metal/intermetallic diffusion couples as we were required to find a second source to fabricate the diffusion couples. The  $\text{W}/\text{Ni}_3\text{Al}$  couples have recently been completed, and we are currently annealing the samples. The  $\text{TiAl}/\text{Ni}_3\text{Al}$  couples are currently being hot-pressed. We expect that these couples will be completed by the end of January.

Since the diffusion path of the  $\text{TiAl}/\text{Ni}_3\text{Al}$  diffusion couples is expected to run through the H-phase region ( $\text{TiNi}_2\text{Al}$ ) of the Ni-Al-Ti phase diagram, we are also having diffusion couples of  $\text{NiTi}/\text{Ni}_3\text{Al}$  made. This will allow us to very simply experimentally determine the ternary diffusion coefficients of the H-phase, thereby providing both verification of the analytical techniques to be employed for the  $\text{Ni}_3\text{Al}/\text{TiAl}$  system, and information that may be useful to those interested in monolithic  $\text{TiNi}_2\text{Al}$  or  $\text{TiNi}_2\text{Al}$  precipitate strengthened  $\text{NiAl}$ .

### III. LECTURES AND PUBLICATIONS

#### A. LECTURES

"Development of High Temperature Composites with long term Stability: Intermetallics and Diffusion Barriers," J.K. Tien, M.W. Kopp, A.J. Carbone, and R.S. Bellows, Industry-University Advanced Materials Conference, Denver CO, 6-9 March 1989.

"Development of High Temperature Composites: Diffusion Barriers," J.K. Tien and M.W. Kopp, 1988 AFWAL High Temperature Materials Workshop, Dayton OH, 13-14 December 1988.

"Reaction Kinetics Between Fiber and Matrix Components in Metal Matrix Composites," M.W. Kopp, J.K. Tien, and D.W. Petrasek, 6th International Symposium on Superalloys, Champion PA, 19-22 September 1988.

"Interdiffusional Instability in High Temperature Metal Matrix Composites," M. W. Kopp and J.K. Tien, 9th International RISO Symposium, Roskilde, Denmark, 5-9 September, 1988.

#### B. PUBLICATIONS

J.K. Tien, M.W. Kopp, A.J. Carbone, and R.S. Bellows, "Development of High Temperature Composites with long term Stability: Intermetallics and Diffusion Barriers," Proc. Industry-University Advanced Materials Conference, Denver CO, 6-9 March 1989, to be published.

M.W. Kopp, J.K. Tien, and D.W. Petrasek, "Reaction Kinetics Between Fiber and Matrix Components in Metal Matrix Composites," Proc. 6th International Symposium on Superalloys, Champion PA, 19-22 September 1988, The Metallurgical Society, (1988) 193-201.

M.W. Kopp and J.K. Tien, "A Preliminary Study of Ion Implanted Diffusion Barriers in High Temperature Metal Matrix Composites," Scripta Met., 22 (9) (1988) 1527-30.

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